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IS 3399 (1993): Zinc oxide for rubber industry [PCD 13: Rubber and Rubber Products]

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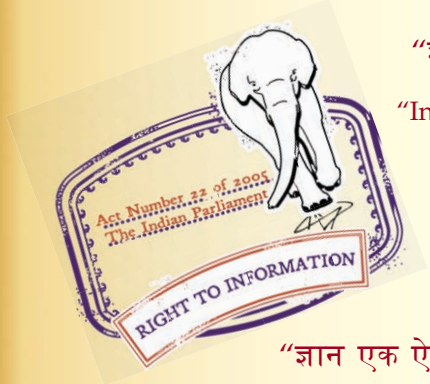

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"Knowledge is such a treasure which cannot be stolen"

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IS 3399 : 1993
(Reaffirmed 2003)

भारतीय मानक

रबड़ उद्योग के लिए जिंक आक्साइड — विशिष्टि

(दूसरा पुनरीक्षण)

Indian Standard

ZINC OXIDE FOR RUBBER INDUSTRY — SPECIFICATION

(Second Revision)

UDC 661·847·12 : 678·4·04

BUREAU OF INDIAN STANDARDS
MANAK BHAVAN, 9 BAHADUR SHAH ZAFAR MARG
NEW DELHI 110002

June 1993

Price Group 2

FOREWORD

This Indian Standard (Second Revision) was adopted by the Bureau of Indian Standards, after the draft finalized by the Rubber Products Sectional Committee had been approved by the Petroleum, Coal and Related Products Division Council.

The first revision of this standard was published in 1973. In this revision, classification of zinc oxide has been introduced based on the manufacturing process. Further the requirements of colour, nitrogen surface area and bulk density have been added.

For the purpose of deciding whether a particular requirement of this standard is complied with, the final value, observed or calculated, expressing the result of a test or analysis, shall be rounded off in accordance with IS 2 : 1960 'Rules for rounding off numerical values (*revised*)'. The number of significant places retained in the rounded off value should be the same as that of the specified value in this standard.

Indian Standard

ZINC OXIDE FOR RUBBER INDUSTRY — SPECIFICATION

(Second Revision)

1 SCOPE

1.1 This standard prescribes the classification, requirements and methods of sampling and tests of zinc oxide for use in rubber industry.

2 REFERENCES

The following Indian Standards are necessary adjuncts to this standard:

IS No.	*Title*
265 : 1987	Specification for hydrochloric acid (*third revision*)
1070 : 1992	Reagent grade water (*third revision*)
7086 (Part 1) : 1973	Methods of sampling and test for rubber compounding ingredients, Part 1
12076-1986	Precipitated silica for rubber industry

3 CLASSIFICATION

3·0 The type of zinc oxide used in the rubber industry and related to the production process.

3.1 French Process or Indirect Type

When zinc oxide is manufactured by the burning of zinc vapour (produced by boiling zinc metal) it is called French Process or Indirect type. It is characterized by a high degree of chemical purity. The particle shape is nodular and the size is 0·2-0·5 micron.

3.2 Secondary Zinc Oxide Type

When zinc oxide is manufactured as a by-product of chemical reaction or results from burning zinc vapour produced from die cast scrap zinc, galvanized zinc dross, it is called as secondary zinc oxide. The particle shape is nodular and the size is 0·2-0·7 micron.

4 REQUIREMENTS

The material shall comply with the requirements given in Table 1 when tested according to the procedures given in col 5 of the table.

5 PACKING AND MARKING

5.1 Packing

5.1.1 The material shall be packed in suitable package as agreed to between the purchaser and the supplier.

5.2 Marking

5.2.1 Each package shall be marked with:

a) Name and grade of material,

b) Indication of the source of manufacture,

c) Net mass of the material,

d) Month and year of manufacture, and

e) Lot/Batch number.

5.2.2 Each package may also be marked with the Standard Mark.

6 SAMPLING

6.1 The representative samples of the material shall be drawn as prescribed in IS 7086 (Part 1) : 1973.

6.2 Number of Tests

Tests for determination of manganese and copper shall be conducted on individual samples. Tests for all other characteristics shall be conducted on the composite sample.

6.3 Criteria for Conformity

6.3.1 *For Individual Samples*

The mean and range of the test results for manganese and copper shall be calculated as follows:

$$\text{Mean } (X) = \frac{\text{The sum of test results}}{\text{No. of the test results}}$$

Range (R) = The difference between the maximum and the minimum value of the test results.

The lot shall be deemed to have satisfied the requirements of the specification if,

$X + 0{\cdot}6\,R \leqslant 0{\cdot}003$ for manganese and
$\leqslant 0{\cdot}001$ for copper

6.3.2 *For Composite Sample*

In respect of all other characteristics, the lot shall be considered as conforming to the specification if the composite sample satisfies each of these requirements.

7 TEST METHODS

7.1 Tests shall be conducted according to the methods prescribed in Annex A. Reference

Reference to relevant clauses of IS 7086 (Part 1) : 1973 are given in col 5 of Table 1.

7.2 Quality of Reagents

Unless specified otherwise pure chemicals and distilled water (*see* IS 1070 : 1992) shall be employed in tests.

NOTE — 'Pure chemicals' shall mean chemicals that do not contain impurities which affect the results of analysis.

Table 1 Requirements of Zinc Oxide for Rubber Industry

(*Clauses* 4 *and* 7.1)

Sl No.	Characteristic	Requirements		Methods of Test, Ref to Cl of IS 7086 (Part 1) : 1973/ Annex
		French Process Indirect Type	Secondary Type	
(1)	(2)	(3)	(4)	(5)
i)	Colour	White powder	Light yellow	Visual
ii)	Zinc oxide (as ZnO), on dried sample at 105°C, percent by mass, *Min*	99·5	99·0	A
iii)	Matter insoluble in hydrochloric acid, percent by mass, *Max*	0·15	0·15	9
iv)	Moisture content, percent by mass, *Max*	0·25	0·25	7
v)	Sieve residue, percent by mass, *Max*			
	a) through 45-micron IS sieve	0·15	0·15	3
	b) through 75-micron IS sieve	0·05	0·05	3
vi)	Relative density at 27/27°C	5·55 to 5·68	5·55 to 5·68	4
vii)	Nitrogen surface area m^2/g	3·5 to 5.5	3·5 to 5·5	A-8 of IS 1 076 : 1986
viii)	Lead (as Pb), percent by mass, *Max*	0·10	0·10	14
ix)	Copper (as Cu), percent by mass, *Max*	0·001	0·001	12
x)	Manganese (as Mn), percent by mass, *Max*	0·003	0·003	11
xi)[1]	Bulk density, g/cc	← To meet customer requirement →		

[1] Bulk density of zinc oxide made by American process will be typically in the range of 820 to 950 g/cc and bulk density of zinc oxide made by French process will be typically between 450 to 550 g/cc.

ANNEX A

[*Clause* 7.1, and *Table* 1, *Sl No.* (ii)]

DETERMINATION OF ZINC OXIDE

A-1 REAGENTS

A-1.1 Dilute Hydrochlorie Acid — 4 N.

A-1.2 Dilute Ammonium Hydroxide Solution — 4 N.

A-1.3 Congo Paper

A-1.4 Standard Zinc Chloride Solution

0·5 percent (*m/v*) (as Zinc). Weigh accurately 5·0 g of chemically pure zinc, dissolve in 300 ml of 4 N hydrochloric acid, and dilute with water to 1 000 ml in a graduated flask.

A-1.5 Diphenylamine Solution

5 percent (*m/v*).

A-1.6 Standard Potassium Ferrocyanide Solution

Approximately 0·05 m. Dissolve 21·0 g of potassium ferrocyanide, 300 mg of potassium ferricyanide and 2 g of anhydrous sodium carbonate (to stabilize the solution) in water, and dilute with water to 1 000 ml.

A-1.7 Concentrated Hydrochloric Acid

Relative density 1·18 (conforming to IS 265 : 1989).

A-1.8 Hydrogen Sulphide Solution

Saturated aqueous solution.

A-1.9 Lead Acetate Paper

A-1.10 Hydrogen Peroxide

3 percent solution.

A-1.11 Ammonium Hydroxide

Relative density 0·92.

A-2 PROCEDURE

A-2.1 Standardization of the Potassium Ferrocyanide Solution

Take 25·0 ml of zinc chloride solution by means of a pipette and dilute ammonium hydroxide solution until a piece of congo paper touched on to the solution just turns pure red colour. Then carefully neutralize the solution with dilute hydrochloric acid from a dropping bottle. Add a few drops in excess until the congo paper turns to a red-blue or a blue-red colour (*p*H 1·5 to 3·0). Make up to 150 ml with water, heat the solution to boiling and add 10 drops of diphenylamene solution. Immediately titrate the solution with potassium ferrocyanide solution until the colour turns to a lasting yellow or yellowish-green (T_1 ml being used). Then back titrate the solution with zinc chloride solution until the colour just turns to blue again (T_2 ml being used).

The standardization factor (f) of the potassium ferrocyanide solution in g of zinc/ml is:

$$f = \frac{0{\cdot}005\ (25 + T_2)}{T_1}$$

A-2.2 Weigh accurately about 1·0 g of the material, previously dried to constant mass at 105 ± 2°C and evaporate to dryness with a mixture of 15 ml of concentrate hydrochloric acid and 30 ml of distilled water. Dissolve the residue by gentle heating, if necessary, add 7 ml of concentrated hydrochloric acid and 30 ml of water, then add 75 ml of hydrogen sulphide solution and heat the suspension to a temperature of 40°C. Allow to stand for one hour at this temperature. When the lead sulphide has settled, filter the liquid and wash the residue thoroughly with a mixture of 25 ml of hydrogen sulphide solution, 5 ml of concentrated hydrochloric acid and 75 ml of water. Boil the filtrate and washings to expel hydrogen sulphide (test on lead acetate paper). After cooling (*see* **A-2.2.1**) the solution, make up to 500 ml in a graduated flask with distilled water. Remove 100 ml of this solution by means of a pipette and add to this ammonium hydroxide until a piece of congo paper, added to the solution, just turns to a pure red colour. Neutralize the solution carefully with hydrochloric acid from a dropping bottle, add a few drops in excess until the congo paper turns to a red-blue or blue-red colour (*p*H 1·5 to 3·0). Heat the solution to boiling, add 10 drops of the diphenylamine solution, and immediately titrate the solution in a similar manner as described for the standardization of the potassium ferrocyanide solution in **A-2.1** (T_3 ml of the potassium ferrocyanide solution and T_4 ml of the zinc chloride solution being required.

A-2.2.1 If the solution contains iron or manganese, add, after cooling about 1 ml of 3 percent hydrogen peroxide and 60 ml of concentrated ammonia. Make up to 500 ml with distilled water and allow the solution to stand for two hours, then filter through an absolutely dry filter paper and funnel. Discard the first 10 to 20 ml of the filtrate. Collect the remainder in a dry flask, remove 100 ml by means of a pipette and boil this to expel ammonia. Then acidify this solution with 4 N hydrochloric acid until a piece of corgo paper turns red-blue or blue-red (*p*H 1·5 to 3·0). Heat the solution to boiling, add 10 drops of the diphenylamine solution and immediately titrate the solution in a similar manner as described for the standardization of the potassium ferrocyanide solution in **A-2.1** (T_3 of the potassium ferrocyanide solution and T_4 ml of the zinc chloride solution being used).

A-3 CALCULATION

$$\text{Zinc oxide, percent by mass} = \frac{622{\cdot}3\ (f\,T_3 - 0{\cdot}005\,T_4)}{M}$$

where

f = standardization factor of the potassium ferrocyanide solution,

T_3 = volume in ml of standard potassium ferrocyanide solution,

T_4 = volume in ml of zinc chloride solution, and

M = mass in g of dry material taken for the test.

Standard Mark

The use of the Standard Mark is governed by the provisions of the *Bureau of Indian Standards Act, 1986* and the Rules and Regulations made thereunder. The Standard Mark on products covered by an Indian Standard conveys the assurance that they have been produced to comply with the requirements of that standard under a well defined system of inspection, testing and quality control which is devised and supervised by BIS and operated by the producer. Standard marked products are also continuously checked by BIS for conformity to that standard as a further safeguard. Details of conditions under which a licence for the use of the Standard Mark may be granted to manufacturers or producers may be obtained from the Bureau of Indian Standards.

Bureau of Indian Standards

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